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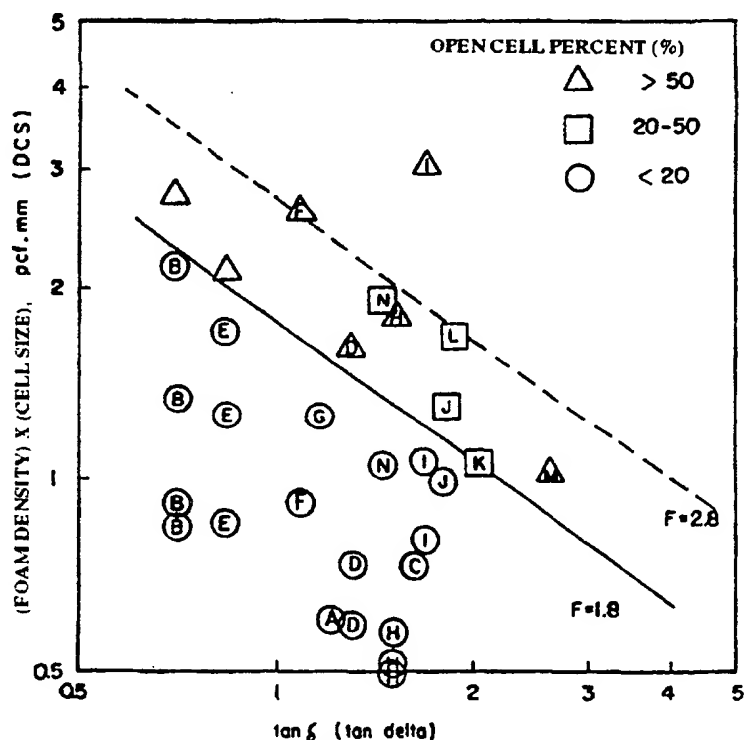## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: EXTRUDED CLOSED-CELL PROPYLENE POLYMER FOAM AND METHODS OF MAKING THE SAME

(57) Abstract

A closed-cell propylene polymer foam, with at least eighty percent closed cells and a foamability characteristic of less than 1.8 is disclosed. Also disclosed is a method for making the closed-cell propylene polymer foams.

EXTRUDED CLOSED-CELL PROPYLENE POLYMER FOAM
AND METHODS OF MAKING THE SAME

The present invention relates to extruded
closed-cell propylene polymer foam. The present
5 invention also relates to several methods for making
this extruded closed-cell propylene polymer foam.

The polypropylene foam of the present invention
has uniform and fine closed cell structure.
10

Since polypropylene generally has a high
crystallinity and a low melt viscosity and is difficult
to cross-link, it has hitherto been quite difficult to
obtain a highly expanded product therefrom. One such
15 kind of product is radiation-crosslinked foam-sheet.

However, since polypropylene is superior to
polyethylene in heat resistance, polypropylene is
expected to become useful in the field requiring a heat
20 resistance of 100 degrees centigrade or higher, such as
heat-insulating material for hot water piping, if a
highly expanded product can be obtained therefrom with
relative inexpensiveness by the non-crosslinking
extrusion foaming process similarly to polyethylene.
25

Also, the foamed closed-cell polypropylene products of the present invention have good cushioning properties at low densities.

Closed-cell polypropylene foams, like
5   polyethylene foams, could be widely employed in the field of packaging in order to provide cushion properties. Fragile objects intended to be transported may be encased and supported in a closed cell foamed polypropylene cushion adapted to conform to the external
10 shape of the object for which protection is desired. One consideration when providing cushioning is shipping costs. Larger thicknesses of cushioning foam result in excess packaging sizes and concomitant elevated shipping
15 costs. So it is desirable to reduce density of the cushioning material to a minimum while retaining the necessary cushioning properties.

In United States Patent 4,522,955 (Great
20 Britain 2,099,434) there is disclosed an extrusion process for highly expanded polypropylene resin foams. These specific polypropylene resin foams use a polypropylene resin having at a stated temperature a minimum value of melt tension and a stated upper limit
25 for a maximum/minimum ratio of melt tension. More specifically, for this non-crosslinking extrusion process, in addition to the ingredients of polypropylene resin and volatile foaming agent, it is required that a nucleus-forming agent, such as inorganic substances,
30 carbon dioxide generators, inorganic or organic acids and thermal type decomposition type chemical foaming agents, be included as part of the mixture to form the polypropylene resin foams of this disclosure.

The present invention is directed to extruded closed-cell propylene polymer resin foams, produced with and without a nucleus-forming agent.

5   The present invention is also directed to extrusion methods of making these closed-cell propylene polymer resin foams with and without a nucleus-forming agent.

10   The present invention is a closed-cell foamed propylene resin having at least eighty percent (80 percent) closed-cells and a foamability characteristic of less than 1.8.

15   The present invention also includes processes for making such closed-cell foamed propylene resin. The process consists of feeding a propylene polymer resin, having a melt flow index less than 10 decigrams/minute at 230 degrees centigrade into a continuous melting device to form a propylene polymer resin melt, then

20   adding the volatile foaming or blowing agent(s) into the melted propylene polymer resin, uniformly mixing the blowing agent and melted resin to form a mixture, cooling the mixture to a (foam) forming temperature,

25   discharging the mixture into a zone of lower pressure and forming the closed-cell foamed propylene resin product having a foamability characteristic (F) of less than or equal to 1.8.

30   A similar method for producing a closed-cell lightly crosslinked foamed propylene resin includes the step of feeding azido functional silanes, vinyl functional silanes, organic peroxides, multifunctional vinyl monomers or mixtures of these compounds as a crosslinking agent into the melted resin.

Figure 1 shows the relationship between foam density, cell size and tan delta for making a closed-cell foam using the resins listed in Table Ia.

5   The closed-cell propylene polymer resin foams of the present invention are those foams having a density of less than 5 pounds per cubic foot. Preferably these foams have a cell size less than 4 millimeters (mm) greater than 0.05 mm.

10   These foams may be prepared in a conventionally known manner by characterized by feeding, into an extruder, a propylene polymer resin having a melt flow index less than 10 decigrams/minute optionally adding a nucleating agent, melting and plasticizing the propylene 15 polymer resin, thereafter feeding a volatile foaming agent, uniformly mixing the materials, and then extruding the mixture to a low pressure zone while cooling it and forming a propylene polymer resin foam 20 product.

Others suitable processes are a coalesced foam process as described in United States Patent Number 4,824,720 and an accumulating extrusion process 25 described in United States Patent Number 4,323,528.

United States Patent Number 4,824,720 describes a method for providing a closed cell foam structure comprising a plurality of coalesced extruded strands or 30 profiles by extrusion foaming of a molten thermoplastic composition utilizing a die containing a multiplicity of orifices. The orifices are so arranged such that the contact between adjacent streams of the molten extrudate occurs during the foaming process and the contacting surfaces adhere to one another with sufficient adhesion

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to result in a unitary structure. The individual strands
of coalesced polyolefin foam should remain adhered into
a unitary structure to prevent strand delamination under
stresses encountered in preparing, shaping, and using
the foam.

5

United States Patent Number 4,323,528 describes
an accumulating extrusion process. In this accumulating
extrusion process low density, elongated cellular bodies
having large lateral cross-sectional areas are prepared
10 by: 1) forming, under pressure, a mixture of a
thermoplastic polymer and a blowing agent, with the
mixture having a temperature at which the viscosity of
the mixture is sufficient to retain the blowing agent
15 when the mixture is allowed to expand; 2) extruding the
mixture into a holding zone maintained at a temperature
and pressure which does not allow the mixture to foam,
the holding zone having an outlet die defining an
orifice opening into a zone of lower pressure at which
20 the mixture foams, and an openable gate closing the die
orifice; 3) periodically opening the gate; 4)
substantially concurrently applying mechanical pressure
by a movable ram on the mixture to eject the mixture
from the holding zone through the die orifice into the
25 zone of lower pressure, at a rate greater than that at
which substantial foaming in the die orifice occurs and
less than that at which substantial irregularities in
cross-sectional area or shape occurs; and 5) permitting
30 the ejected mixture to expand unrestrained in at least
one dimension to produce an elongated thermoplastic
cellular body.

As used herein, "propylene polymer material"
means propylene polymer material selected from the group
consisting of (a) homopolymers of propylene, (b) random

and block copolymers of propylene and an olefin selected
from the group consisting of ethylene, C^4-C^{10} 1-olefins,
and C^4-C^{10} dienes, provided that, when said olefin is
ethylene, the maximum polymerized ethylene content is
less than 20 percent by weight, when said olefin is a
5   C^4-C^{10} 1-olefin, the maximum polymerized content thereof
is less than 20 percent by weight, and when said olefin
is a C^4-C^{10} diene, the maximum polymerized content
thereof is less than 20 percent by weight, and (c)
10   random terpolymers of propylene and 1-olefins selected
from the group consisting of ethylene and C^4-C^8 1-
olefins, provided that the maximum polymerized C^4-C^8 1-
olefin content is less than 20 percent by weight, and
when ethylene is one of said 1-olefins, the maximum
15   polymerized ethylene content is less than 20 percent by
weight. The C^4-C^{10} 1-olefins include the linear and
branched C^4-C^{10} 1-olefins such as, for example, 1-
butene, isobutylene, 1-pentene, 3-methyl-1-butene, 1-
hexene, 3,4-dimethyl-1-butene, 1-heptene, 3-methyl-1-
20   hexene. Examples of C^4-C^{10} dienes include 1,3-
butadiene, 1,4-pentadiene, isoprene, 1,5-hexadiene, 2,3-
dimethyl-1,3-hexadiene. The propylene polymer material
must have at least eighty percent (80 percent) percent
25   polypropylene in the propylene polymer material.

Apart from the above, other polymeric
substances with the above-mentioned polypropylene type
resins, such as high- and low-density polyethylenes,
30   polybutene-1, ethylene-vinyl acetate copolymer,
ethylene-propylene rubber, styrene-butadiene rubber,
ethylene-ethyl acrylate copolymer, ionomer, may be mixed
into the above-mentioned polypropylene type resins
either alone or in the form of a blend of two or more
polymers, so long as polypropylene is the main component

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in the resulting mixture and the mixture is of uniform quality.

Also, as used herein, the propylene polymer material has a weight average molecular weight of at
5   least 100,000.

The preferred propylene polymer resins for the present invention are those propylene polymer resins which are branched or lightly cross-linked polymer
10   materials. Branching (or light cross-linking) may be obtained by those methods generally known in the art, such as chemical or irradiation branching/light cross-linking. One such resin which is prepared as a branched/lightly cross-linked propylene polymer resin
15   prior to using the propylene polymer resin to prepare a finished propylene polymer resin product and the method of preparing such a propylene polymer resin is described in United States Patent Number 4,916,198. Another
20   method to prepare branched/lightly cross-linked propylene polymer resin is to introduce chemical compounds into the extruder, along with a propylene polymer resin (preferably unbranched and non-lightly cross-linked) and allow the branching/lightly cross-
25   linking reaction to take place in the extruder. United States Patent Number 4,714,716 illustrates this method.

A propylene polymer material or blend of propylene polymer materials is melt processed in a
30   conventional manner by feeding, melting, and metering it in a conventional melt processing apparatus such as an extruder. A volatile blowing agent and an optional crosslinking agent are mixed with the propylene polymer material under pressure to form a flowable gel or admixture. The crosslinking agent, if added, is added

in an amount which is sufficient to initiate crosslinking and raise the pressure of the gel or admixture to less than that pressure which causes melt fracture of the polymer to occur. "Melt fracture" is a term used in the art to describe a melt flow instability of a polymer as it is extruded through a die which flow instability causes voids and/or other irregularities in the final product.

Suitable branching/crosslinking agents for use in extrusion reactions have been found to include azido and vinyl functional silanes, organic peroxides and multifunctional vinyl monomers.

It is also possible to add various additives such as inorganic fillers, pigments, antioxidants, ultraviolet absorbers, flame retardants, processing aids.

One additive which is not necessary but is generally desirable in some circumstances, is a nucleating agent as is generally known in the art.

As a nucleating agent optionally used in this invention for controlling dispersion and size of cells, there can be referred to inorganic substances such as calcium carbonate, talc, clay, titanium oxide, silica, barium sulfate, diatomaceous earth ; carbon dioxide generator by the combination of a bicarbonate or a carbonate of sodium, potassium, ammonium or the like and an inorganic or organic acid such as boric acid, citric acid, tartaric acid or the like; thermal decomposition type chemical foaming agents such as azodicarbonamide, benzenesulfonyl hydrazide, toluenesulfonyl hydrazide; and so on. Among them, finely pulverized products of

inorganic substances such as calcium carbonate, talc are
relatively preferable in that they have no restriction
as to extrusion temperature.

5   The amount of said nucleating agent may be
appropriately selected from the range of 0.01-5 parts by
weight per 100 parts by weight of polypropylene resin.
In this range, the most preferable amount is 0.1-3 parts
by weight. In general, an increased amount of
nucleating agent gives a smaller diameter of cell.

10 However, if the amount exceeds 5 parts by weight,
agglomeration or insufficient dispersion of nucleating
substance occurs, so that the diameter of the cell
becomes greater. On the contrary if the amount is less

15 than 0.01 part by weight, the nucleating action is too
feeble to decrease the diameter of the cells.

      The volatile foaming agents usable in this
invention generally have a boiling point temperature

20 range of -50°C to +50°C and include, but are not limited
to aliphatic hydrocarbons such as n-pentane, iso-
pentane, neo-pentane, isobutane, n-butane, propane;
fluoro-chlorinated hydrocarbons such as
dichlorotetrafluoroethane, trichlorotrifluoroethane,

25 trichloromonofluoromethane, dichlorodifluoromethane,
dichloromonofluoromethane; and so on. Among them, the
non-fully halogenated hydrocarbons are preferable in
point of environmental considerations. Particularly
preferred among the non-fully halogenated hydrocarbons

30 are partially or fully fluorinated hydrocarbons and non-
fully halogenated
fluoro-chlorinated hydrocarbons. Examples of these
include 1-chloro-1,1-fluoroethane and
1,1-difluoroethane. Particularly preferred among the
aliphatic hydrocarbons is isobutane and

isobutane/n-butane mixtures. Also contemplated are
combinations of these blowing agents with minor amounts
of CO_2, H_2O, N_2 and argon in the mixtures. Also
decomposable blowing agents, such as azobisformamide,
may be incorporated with the volatile foaming agents.

5   Mixtures of any or all of these volatile foaming agents
are also contemplated within the scope of this
invention.

From the viewpoint of foaming workability and

10 quality of foamed product, the amount of foaming agent
added to the propylene polymer resin is preferably in
the range of 0.05-0.5 pound/moles by weight per 100
pounds by weight of propylene polymer resin. Typically,

15 the amount of foaming agent incorporated into the
propylene polymer resin depends on the desired foam
density. In general, incorporation of a greater amount
of foaming agent results in a higher expansion ratio
(the term "expansion ratio" herein referred to means the

20 ratio (density of resin)/(density of expanded product))
and thus a lower foam density. However care must be
taken not to incorporate an amount of foaming agent that
causes a separation between resin and foaming agent in
the extruder. When this happens, "foaming in the die"

25 occurs, the surface of the expanded product becomes
rough and no good expanded product is obtainable.

The propylene polymer foam of the present
invention is a closed-cell foam, which means that for

30 the purpose of the present invention eighty percent or
greater of the cells are closed. Closed-cell content is
measured in any conventionally known manner.

Surprisingly it has been found that a
combination of propylene polymer resin properties and

final propylene polymer foam product properties are
required to produce the closed cell propylene polymer
foam of the present invention.

5   The foamability factor is the combination of
these properties. This factor is expressed by the
following equation:

$$F = \rho_f * D * (\tan \delta)^{0.75} \leq 1.8 \quad \text{(Equation 1)}$$

10   where   F is the foamability factor

ρ_f is the density of the foam in pounds
per cubic foot;

15   D is the average cell size diameter in
millimeters;

$\tan \delta$ is g"/g'; where
g" is the loss modulus; and
20   g' is the storage modulus of the
polymer melt using 2.5 mm thick and
25 mm diameter specimens at 190° C
(centigrade) at one radian per
25   second oscillating frequency.

The foamability factor must be less than or
equal to 1.8 for the foam to have at least eighty
percent closed-cells. Preferably the foamability factor
30 is less than or equal to 1.5.

The parameter $\tan \delta$ may be determined by using a
mechanical spectrometer, such as model RMS-800,
available from Rheometrics, Inc. in Piscataway, New
Jersey, United States of America.

In the evaluation of rheological
characteristics, such as tan δ, g' and g", of
visoelastic materials, such as polymer melts, a
disk-like specimen, measuring 2.5mm in thickness and
25 mm in diameter is placed between opposed, axially
5   spaced apart, radially-extending surfaces and is coupled
to each surface, filling the axial spacing between the
surfaces. One of the surfaces then is rotated the
axial direction relative to the other to place the test
10   specimen in shear and the torque resulting from the
shear is measured. The shear may be steady shear, in
which case the measured torque is constant, or the shear
may be dynamic shear, in which case the measured torque
changes continuously with time. The measured torque is
15   proportional to the viscous, or loss component of the
modulus (g") of the material. For the purpose of this
invention the shear is steady shear, meaning the
measured torque, and thus g", is constant at the given
temperature. As a result of the nature of the forces
20   applied to the test specimen in this procedure, the test
specimen has a tendency to expand axially, thereby
placing axially directed forces upon the relatively
rotating surfaces to which the specimen is coupled.
25   This axial force exerted upon the surfaces by the test
specimen under shear conditions is proportional to the
elastic, or storage component of the modulus (g') of the
material. The parameter tan δ for the present invention
is then calculated as g" divided by g' at the stated
30   temperature and oscillating frequency.

In addition to meeting the requirements of
Equation 1, the mixture of the blowing agent and
propylene polymer material (foaming gel) must be cooled
down to the optimum foaming temperature, which is easily

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determined experimentally. That is, the foam meeting
the requirement as described by Equation 1 does not
necessarily end up with a closed cell foam if the
foaming gel is not optimally cooled. In other words,
Equation 1 is a necessary but not a sufficient condition
to produce a closed-cell polypropylene foam.

5

Dimensional stability is also an important
factor for closed-cell foams. In the most desirable
case the foam would not shrink at all or if it did would
recover its original volume in a period of minutes or
hours. Thus 'foam dimensional stability' (hereafter
called DS) is an important criterion for formulation
acceptability.

10

15

For the purpose of this specification a foam
has satisfactory DS as long as its dimensions recover
substantially to the values listed here.

Dimensionally stable foam - a foam that shrinks no more
                           than 50 percent (minimum
                           foam value>50 percent) and
                           recovers to over 75 percent
                           of the initial within a
                           week and to over 85 percent
                           of the initial within six
                           months,

20

25

Preferably   - the foam shrinks no more
                           than 35 percent and
                           recovers to over 85 percent
                           within a week

30

Most preferably   - the foam shrinks no more
                           than 20 percent and
                           recovers to over 90 percent
                           within a week.

These percentage volume numbers are based on a base foam volume of the foam measured within three minutes of exit from the die.

5   Another condition that is preferable, but not required is that there be forming plates, consisting of two flat surfaces which are lubricated or covered with a material, such a polytetrafluoroethylene, to reduce friction and prevent sticking, after the die to
10 constrain the foam exiting from the die. The use of such forming plates increases the foam cross-section and cell size and produces a foam product has an unfolded and smooth surface appearance. Surprisingly while the forming plates worked well for the closed-cell
15 polypropylene polymer material foam, the forming plates caused a ragged surface and an increase in density when used to make a low density polyethylene foam.

20

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30

The following Examples illustrate the present
invention.

Example I

5   The equipment use in this example is a 1 inch
(2.5 centimeters (cm)) screw type extruder having
additional zones of mixing and cooling at the end of
usual sequential zones of feeding, metering, and mixing.
An opening for blowing agent is provided on the extruder

10   barrel between the metering and mixing zones. At the
end of the cooling line, there is attached a die orifice
having an opening of rectangular shape. The height of
the opening, hereinafter called die gap, is adjustable

15   while its width is fixed at 0.145 inch (3.68 mm
(millimeter)).

        Granular polypropylene resin was selected from
the group presented in Table Ia.

20

25

30

Resin Designation	Resin Type (1)	Resin Class. (2)	M.F.R. (dg/min) (3)	tan δ (4)	Remarks (5)
A	H	C	0.2	1.22	Profax 6823
B	H	HMS	0.25	0.71	Escorene PD-5291
C	BC	C	0.4	1.63	Profax 7823
D	RC	C	0.5	1.31	Developmental
E	H	HMS	0.5	0.84	Developmental
F	H	HMS	0.65	1.09	Developmental
G	RC	HMS	0.8	1.17	Developmental
H	RC	C	1.5	1.51	7235 by Fina
I	RC	C	2	1.69	7300 MF by Quantum
J	RC	C	3	1.83	7300 KF by Quantum
K	RC	C	5	2.04	3303 Gk by Quantum
L	RC	HMS	6.7	1.88	Developmental
M	RC	C	7	2.64	Dypro W-756
N	RC	HMS	8	1.46	Developmental

Notes:

(1) H = homopolymer, BC = block copolymer of 5-10 percent ethylene and 90-95 percent propylene, RC = random copolymer of 2-4 percent ethylene and 96-98 percent propylene

(2) C = conventional resins, HMS = high melt strength resins

(3) Melt flow rate determine per ASTM D 1238 Condition L

(4) tan δ = loss modulus/storage modulus determined on a
model RMS-800 mechanical spectrometer by Rheometric,
Inc. with having 2.5 mm thickness and 25 mm diameter
specimens on parallel plates at 190°C at one radian
per second oscillating frequency

(5) Profax = Trademark of Himont United StatesA., Inc.
5   Escorene = Trademark of Exxon Chemical Americas
Fina = Final Oil & Chemical Co.
Quantum = Quantum Chemical Corp.
Dypro = Trademark of Arco Chemical Co.
Developmental = developmental resins made by Himont
United StatesA., Inc.

10

     The granular polypropylene resin was fed into
the extruder at a uniform rate of approximately 4 pounds
per hour (lbs/hr) (1.8 kilograms per hour (kgh)). The
15 temperature maintained in the extruder zones were 130°C
(centigrade) at feeding zone, 190°C at melting zone,
210°C at metering zone and 220°C at the mixing zone. A
predetermined blowing agent mixture consisting of HCFC-
142b (1-chloro-1,1-difluoroethane) and EtCl (ethyl
20 chloride) was prepared in a weigh tank and injected
under pressure into the mixing zone at a predetermined
uniform rate which ranged from 0.16 lb-moles (pound-
moles) of blowing agent mixture per one hundred lbs
(pounds) of polymer (abbreviated mph hereinafter) to
25 0.28 mph. For each polymer, the foaming test was
started with a high level of blowing agent and the level
was reduced down to the lower levels of blowing agent.
For some tests, a small amount of talcum powder was
30 mixed in the polymer for cell size control. The
temperature of the cooling zone was maintained so that
the polymer/blowing agent mixture could reach an optimum
foaming temperature. The foaming temperature varied
widely among the polymers and ranged from 130°C to 156°C.

Once a best foaming condition was reached for the given formulation, the die gap was adjusted to provide a foam strand free from prefoaming. This foam strand had a smooth skin. At the threshold die gap for prefoaming, foam samples were saved and aged at ambient temperature. Foam density, cell size, open cell content, and foam cross-sectional area were determined with foam specimens aged for at least one month. Tan delta (tan δ) values were determined with the virgin resins on a model RMS-800 mechanical spectrometer (manufacture by Rheometrics, Inc.) during temperature sweep at one radian per second oscillating frequency. The parallel plates configuration was employed and the test specimens had been molded at 180°C into disks of 2.5 mm in thickness and 25 mm in diameter.

Table Ib summarizes the results of this Example I.

TABLE Ib

Test. No.	Resin Designation	M.F.I. (1)	tanδ (2)	Blowing Ratio (3)	Agent Level (4)	Talc Level (5)	Foam Density (ρΩ)(6)	Cell Size (7)	(ρΩ•D) (8)	Open Cell (9)	Cross-sectional Area (10)
A.1	A	0.2	1.22	60/40	0.22	-	1.39	0.43	0.60	6	0.7
B.1	B	0.25	0.71	60/40	0.22	-	1.34	1.01	1.35	8	1.4
B.2				60/40	0.22	0.03	1.35	0.62	0.84	7	1.0
B.3				60/40	0.19	0.1	1.49	0.62	0.92	9	1.1
B.4				60/40	0.22	-	1.35	2.03	2.74	59	1.94
B.5				60/40	0.22	-	1.35	1.62	2.19	19	1.34
C.1	C	0.4	1.63	60/40	0.28	-	1.46	0.51	0.74	19	0.4
D.1	D	0.5	1.31	60/40	0.28	-	1.12	0.65	0.73	6	0.8
D.2				60/40	0.22	-	1.44	0.41	0.59	6	0.9
*D.3				60/40	0.25	0.2	1.23	1.30	1.60	65	1.5
E.1	E	0.5	0.84	60/40	0.22	-	1.30	0.65	0.85	6	1.0
E.2				60/40	0.16	-	1.60	1.08	1.73	1	1.3
E.3				60/40	0.13	0.2	1.96	0.65	1.27	7	1.1
*E.4				60/40	0.13	-	1.97	1.08	2.13	67	1.5
F.1	F	0.8	1.09	60/40	0.22	-	1.35	0.68	0.92	0	0.7
*F.2				60/40	0.13	-	2.87	0.90	2.58	86	1.1
G.1	G	0.8	1.17	60/40	0.16	0.2	1.74	0.72	1.25	18	0.9
H.1	H	1.5	1.51	60/40	0.19	0.5	1.63	0.31	0.51	8	0.9
H.2				60/40	0.16	-	1.83	0.26	0.48	9	0.9
H.3				60/40	0.16	0.25	1.77	0.32	0.57	5	1.2
*H.4				60/40	0.16	-	2.66	0.68	1.81	87	1.0
I.1	I	2	1.69	60/40	0.22	-	1.32	0.81	1.07	14	1.1
I.2				60/40	0.19	0.3	1.59	0.50	0.80	7	0.8
*I.3				60/40	0.19	-	1.87	1.62	3.03	64	1.4
J.1	J	3	1.83	49/51	0.27	-	1.32	0.74	0.98	1	0.8
J.2				49/51	0.27	-	1.29	1.01	1.30	27	0.7
K.1	K	5	2.04	60/40	0.23	-	1.37	0.77	1.05	26	0.7
L.1	L	6.7	1.88	60/40	0.16	-	1.81	0.93	1.68	28	0.8
*M.1	M	7	2.64	50/50	0.19	-	1.24	0.81	1.00	58	0.5
N.1	N	8	1.46	60/40	0.22	-	1.60	0.65	1.04	14	0.4
N.2				60/40	0.16	-	2.03	0.93	1.89	23	0.5

NOTES: *Not an example of this invention

5   (1) Melt flow index in decigrams per minute determined
            per ASTM D 1238 at Condition L (230°C, 2.16 Kg)

       (2) The ratio of loss modulus to storage modulus of
            polypropylene melt at 190°C and one radian per
            second frequency determined by using a dynamic
            mechanical spectrometer manufactured by
            Rheometrics, Inc. (25 mm diameter x 2.2 mm
10   thickness specimens with parallel plates
            configuration)

       (3) Molar ratio of CFC-142b/EtCl

       (4) G-moles of blowing agent mixed in per hundred
            grams of polymer
15
       (5) Parts of talcum powder mixed in per hundred parts
            of polymer

       (6) Foam density in pounds per cubic foot

       (7) Average cell size in millimeters determined per
20   ASTM D 3576

       (8) Product of foam density and cell size

       (9) Open cell content in percentage determined per
            ASTM D 2856-A

25 (10)   Cross-sectional area of foam body in square
            centimeters

30   Table Ib and Figure 1 indicate that a resin
    having s smaller tan delta is the more readily
    'foamable', that is, the more readily produces closed-
    cell foams. Equation 1 with the A value of 1.8 marks
    well the condition between the closed-cell (open cell
    content< 20 percent) and open-cell foams. Some resins

produce partially open-cell foams (where the open cell content is 20-50- percent) when the foam product meets equation 1 with the A value between 1.8 and 2.8. When the product of density and cell size is greater than foamability (F) of a given resin with the A value of

5   2.8, a substantially open cell foam (open cell content > 50 percent) is produced.

The level or amount of blowing agent also affects the production of a closed cell foam. Generally

10   an increased amount of blowing agent produces a foam having more closed cells. A high level of blowing agent also provides a low foam density and makes the cell size smaller. As the level of blowing agent is decreased,

15   the foam density and cell size become larger requiring the addition of a cell nucleator, such as talc. Even with addition of this nucleator, a closed cell foam is difficult to make when the blowing agent level drops below 0.13 mph.

20

High levels of blowing agent are not always desirable when making a foam product and can produce undesirable characteristics. For example, low density foams made with a high blowing agent level often have

25   small cell sizes and small cross-sectional sizes. In addition, the foam may become corrugated in a transverse direction (as distinguished from the extrusion and vertical directions) because of a rapid expansion rate. Therefore, it is often desirable to use a resin having a

30   small tan delta which allows one to operate at the maximum value of (foam density x cell size) (hereafter called DCS).

Example II

Example I was repeated using only the resin
designated İ in Table Ia.   This resin is made by Quantum
Chemical Co. and is a random propylene-ethylene random
5 copolymer with 2-3 percent ethylene and a melt flow
index of 2.0 as determined using the ASTM D-1238
Condition L standard).

The results of this Example II are shown in
10 Table II.

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TABLE II

Test No.	Blowing Agents Type (1)	Ratio (Mole) (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (pf) (5)	Cell Size (mm) (D) (6)	pf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Dimensional Stability Min. Vol. (%) (10)	1 Wk (%) (11)	7 Mo. (%) (12)
II.1	HCFC-142b/EtCl	100/0	0.28	-	1.25	0.26	0.33	7	0.27	97	98	96
II.2	HCFC-142b/EtCl	100/0	0.25	-	1.31	0.24	0.31	4	0.45	94	97	95
II.3	HCFC-142b/EtCl	100/0	0.22	-	1.31	0.49	0.64	3	0.63	98	99	98
II.4	HCFC-142b/ETCl	100/0	0.19	-	1.53	0.58	0.89	32	0.92	99	99	99
II.5	HCFC-142b/EtCl	90/10	0.28	-	1.18	0.34	0.40	10	0.53	97	98	97
II.6	HCFC-142b/EtCl	90/10	0.25	-	1.24	0.40	0.50	7	0.62	98	98	97
II.7	HCFC-142b/EtCl	90/10	0.22	-	1.29	0.65	0.84	1	0.88	98	100	100
II.8	HCFC-142b/EtCl	90/10	0.19	-	1.46	0.87	1.27	55	1.17	98	99	99
II.9	HCFC-142b/EtCl	80/20	0.28	-	1.22	0.49	0.60	5	0.87	94	97	96
II.10	HCFC-142b/ETCl	80/20	0.25	-	1.27	0.85	1.08	18	0.83	94	98	99
II.11	HCFC-142b/EtCl	80/20	0.22	-	1.44	0.93	1.34	68	0.99	98	98	98
II.12	HCFC-142b/EtCl	70/30	0.28	-	1.13	0.62	0.70	2	0.73	89	100	99
II.13	HCFC-142b/EtCl	70/30	0.25	-	1.26	1.04	1.31	45	1.07	95	99	99
*II.14	HCFC-142b/EtCl	70/30	0.22	-	1.39	1.16	1.61	75	1.35	94	102	97
II.15	HCFC-142b/EtCl	70/30	0.22	0.2	1.35	0.44	0.59	3	0.84	88	98	98
II.16	HCFC-142b/EtCl	70/30	0.19	0.3	1.54	0.46	0.71	2	0.65	91	97	98

TABLE II (CONT.)

Test No.	Blowing Agents Type (1)	Ratio (Mole) (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (pf) (5)	Cell Size (mm) (D) (6)	pf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Dimensional Stability Min. Vol. (%) (10)	1 Wk (%) (11)	7 Mo. (%) (12)
II.17	HCFC-142b/EtCl	60/40	0.31	-	1.22	0.50	0.61	7	0.47	88	100	99
II.18	HCFC-142b/EtCl	60/40	0.25	-	1.23	0.81	1.00	19	0.92	82	96	98
II.19	HCFC-142b/EtCl	60/40	0.22	-	1.32	0.81	1.07	14	1.06	88	95	96
*II.20	HCFC-142b/EtCl	60/40	0.19	-	1.87	1.62	3.03	64	1.38	94	94	95
II.21	HCFC-142b/EtCl	60/40	0.19	0.3	1.59	0.50	0.80	7	0.76	87	97	97
*II.22	HCFC-142b/EtCl	60/40	0.16	0.3	1.91	0.87	1.66	68	0.89	94	95	95
												***
II.23	HCFC-142b/EtCl	50/50	0.37	-	1.02	0.54	0.55	7	0.45	79	96	96
II.24	HCFC-142b/EtCl	50/50	0.34	-	1.07	0.56	0.60	4	0.51	77	96	97
II.25	HCFC-142b/EtCl	50/50	0.31	-	1.09	0.62	0.68	1	0.65	74	94	95
II.26	HCFC-142b/EtCl	50/50	0.28	-	1.19	0.91	1.08	1	0.79	71	92	94
*II.27	HCFC-142b/EtCl	50/50	0.25	-	1.27	1.25	1.59	68	0.94	93	97	98
II.28	HCFC-142b/EtCl	50/50	0.25	0.5	1.41	0.41	0.58	8	0.68	78	95	97
II.29	HCFC-142b/EtCl	50/50	0.22	0.5	1.48	0.43	0.64	5	0.61	76	95	96
II.30	HCFC-142b/EtCl	50/50	0.19	0.5	1.62	0.45	0.73	13	0.63	80	94	95
*II.31	HCFC-142b/EtCl	50/50	0.16	0.5	2.52	0.65	1.64	64	0.49	ND	ND	ND

TABLE II (CONT.)

Test No.	Blowing Agents Type (1)	Ratio (Mole) (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (ρf) (5)	Cell Size (mm) (D) (6)	ρf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Min. Vol. (%) (10)	Dimensional Stability 1 Wk (%) (11)	7 Mo. (%) (12)
II.32	HCFC-142b/EtCl	40/60	0.37	-	1.23	0.72	0.89	8	0.36	75	97	102
II.33	HCFC-142b/EtCl	40/60	0.34	-	1.16	0.96	1.11	9	0.65	65	89	95
*II.34	HCFC-142b/EtCl	40/60	0.31	-	1.13	1.25	1.41	74	0.91	100	102	102
*II.35	HCFC-142b/EtCl	40/60	0.28	-	1.87	1.12	2.09	82	0.69	97	97	97
II.36	HCFC-142b/EtCl	40/60	0.28	0.5	1.46	0.50	0.73	9	0.44	67	88	92
II.37	HCFC-142b/EtCl	40/60	0.25	0.5	1.51	0.46	0.69	6	0.46	65	87	92
II.38	HCFC-142b/EtCl	40/60	0.22	0.5	1.58	0.52	0.82	20	0.52	74	90	93
II.39	HCFC-142b/EtCl	30/70	0.37	-	1.20	0.74	0.89	16	0.52	57	81	87
II.40	HCFC-142b/EtCl	30/70	0.34	-	1.27	0.85	1.08	7	0.60	52	76	83
II.41	HCFC-142b/EtCl	30/70	0.31	-	1.34	0.93	1.25	15	0.72	60	82	90
*II.42	HCFC-142b/EtCl	30/70	0.28	-	1.37	1.35	1.85	67	0.95	92	99	94
II.43	HCFC-142b/EtCl	30/70	0.28	0.3	1.48	0.43	0.64	10	0.41	56	78	86
II.44	HCFC-142b/EtCl	30/70	0.25	0.3	1.64	0.65	1.07	10	0.56	53	75	82
II.45	HCFC-142b/EtCl	30/70	0.22	0.3	1.69	0.54	0.91	21	0.81	60	80	86
*II.46	HCFC-142b/EtCl	30/70	0.19	0.5	1.83	0.65	1.19	63	0.77	70	84	86
*II.47	HCFC-142b/EtCl	30/70	0.16	0.5	2.64	0.81	2.14	80	0.81	92	94	92
II.48	HCFC-142b/EtCl	20/80	0.37	-	1.54	1.01	1.56	23	0.60	38	56	68
II.49	HCFC-142b/EtCl	20/80	0.34	-	1.37	1.20	1.64	24	0.62	41	63	76
*II.50	HCFC-142b/EtCl	20/80	0.31	-	1.27	1.62	2.06	68	0.91	88	94	94
*II.51	HCFC-142b/EtCl	20/80	0.28	-	1.69	1.73	2.92	76	1.12	92	92	92
II.52	HCFC-142b/EtCl	20/80	0.28	0.3	1.70	0.54	0.92	14	0.71	44	66	79
II.53	HCFC-142b/EtCl	20/80	0.25	0.3	1.78	0.54	0.96	18	0.52	47	67	80
II.54	HCFC-142b/EtCl	20/80	0.22	0.4	1.95	0.46	0.90	12	0.49	53	74	83
*II.55	HCFC-142b/EtCl	20/80	0.19	0.5	2.40	0.81	1.94	78	0.72	90	91	91

NOTES: *Not an example of this invention

   (1) Type of blowing agent used

5   (2) Molar ratio

   (3) Pound moles of blowing agent mixed in per hundred
pounds of polymer (corresponding to kilograms per
hundred kilograms of polymer)

   (4) Parts of talcum powder mixed in per hundred parts
10   of polymer

   (5) Density of foam body in pounds per cubic foot
(pcf) measured after size months

   (6) Cell size in millimeters determined per ASTM D
3576
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   (7) Product of density and cell size

   (8) Open cell content in percentage determined per
ASTM D 2856-A

   (9) Cross-sectional area of foam body in square
20   centimeters

  (10) Minimum volume of foam body experiencing during
aging as a percentage of the initial measured
within 30 seconds after extrusion. ND - not
determined.

25 (11), (12) Volume of one week and 7 month old foam,
respectively, as a percentage of the initial
volume measured within 30 seconds after
extrusion

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As can be seen in Table II, there is a general
tendency that the DCS value increases as the EtCl level
in the blowing agent mixture increase. The Quantum
7300MF resin used in this example has tan delta value of
1.69 and thus a DCS value of 1.21 and 1.89 for the F
5   (foamability) values of 1.8 and 2.8, respectively. It
is noted that DCS=1.21 well divides the open cell and
closed cell regions for a resin having a tan delta value
of 1.69 (open cell content < 20 percent). Foams
10 falling between DCS=1.21 and DCS=1.89 are moderately
open-celled (20-50 percent open cell content). Foams
having their DCS values greater than 1.89 are
substantially open-celled (open cell content>50
percent).

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Also from the DS definition stated previously
in the specification and the data in Table II, it can be
seen that a dimensionally stable foam can be made with a
HCFC-142b/EtCl mixture having as little as 30 percent
20 HCFC-142b. Preferably dimensional stability is achieved
with an HCFC-142b level 40 percent or higher. Most
preferably dimensional stability is attained when the
HCFC-142b level exceeds 50 percent. Since the foam
cross-sectional capability decreases as the level of
25 HCFC-142b is increased, the 60/40 and 70/30 by mole
mixture of
HCFC-142b/EtCl provide the most balanced dimensional
stability and foam cross-sectional size.

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Example III

The procedure of Example I was repeated using the same resin designated in Example II.

5   The results are consolidated in Table IIIa for single and binary blowing agent systems and in Table IIIb for HCFC-142b/EtCl/CO2 ternary blowing agents.

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TABLE IIIa

Test No.	Polymer Rate (lb/hr) (12)	Blowing Agent Type (1)	Blowing Agent Ratio (Mole) (2)	Blowing Agent Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (ρf) (5)	Cell Size (mm) (D) (6)	ρf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM²) (9)	Dimensional Stability Min Vol (%) (10)	Dimensional Stability 1 Wk (%) (11)
III.1	3.5	CFC-114	-	0.28	-	1.33	0.10	0.13	6	0.21	92	93
III.2	3.5	CFC-114/EtCl	50/50	0.28	-	1.37	0.70	0.96	13	0.79	95	95
III.3	4	CFC-114/EtCl	30/70	0.28	-	1.25	0.85	1.06	49	1.31	97	99
III.4	4	CFC-114/EtCl	30/70	0.28	0.3	1.36	0.34	0.46	5	0.50	70	110
*III.5	4	CFC-114/EtCl	20/80	0.28	-	1.82	2.31	4.20	79	1.66	92	98
III.6	4.0	CFC-114/EtCl	20/80	0.28	0.2	1.45	0.68	0.99	3	0.99	53	96
*III.7	3.5	EtCl	-	0.28	0.5	1.39	0.72	1.00	33	0.60	28	47
III.8	4	CFC-12	-	0.28	-	1.23	0.48	0.59	7	0.68	71	85
III.9	4	CFC-12/EtCl	50/50	0.28	-	1.43	0.81	1.16	1	1.23	61	80
*III.10	4	CFC-12/EtCl	50/50	0.14	-	2.03	0.95	1.93	75	1.37	ND	ND
*III.11	4	CFC-12/EtCl	20/80	0.28	-	2.05	1.80	3.69	72	1.27	80	90
*III.12	4	CFC-12/EtCl	20/80	0.28	0.3	1.58	0.68	1.07	14	0.72	43	64

Notes: * - Not an example of this invention

(1) through (11) = the same as in Table II
(12) Rate of polymer extruded in pounds per hour

TABLE IIIa (CONT.)

Test No.	Polymer Rate (lb/hr) (12)	Blowing Agent Type (1)	Ratio (Mole) (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (ρf) (5)	Cell Size (mm) (D) (6)	ρf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Dimensional Stability Min Vol. (%) (10)	1 Wk (%) (11)
III.13	4	HCFC-142b/MeCl	80/20	0.28	-	1.07	0.75	0.80	0	0.83	86	97
III.14	4	HCFC-142b/MeCl	50/50	0.28	0.3	1.30	0.54	0.7	2	0.63	75	90
*III.15	4	HCFC-142b/MeCl	30/70	0.28	-	1.37	1.08	1.48	62	1.13	91	96
III.16	4	HCFC-142b/MeCl$_2$	80/20	0.28	-	1.13	0.54	0.61	2	0.67	92	98
*III.17	4	HCFC-142b/MeCl$_2$	50/50	0.28	-	1.49	1.47	2.19	82	0.64	99	87
III.18	4	CFC-12/CFC-11	50/50	0.28	0.3	1.36	0.51	0.69	3	0.76	62	78

Notes: * - Not an example of this invention

(1) through (11) = the same as in Table II
(12) Rate of polymer extruded in pounds per hour

NOTES: (CONTINUED)

CFC-114 =1,2-dichloro-1,1,2,2-tetrafluoroethane

5   CFC-12 = dichlorodifluoromethane

MeCl = methyl chloride

MeCl$_2$ = methylene chloride

CFC-11 = trichlorofluoromethane
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CO$_2$ = carbon dioxide

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TABLE IIIb

Test No.	HCFC-142b/EtCl/CO$_2$		Talc Level (pph)	Foam Density (pcf)	Cell Size (mm) (D)	ρf•D (pcf mm)	Open Cell (%)	Cross-sectional Area (CM2)	Dimensional Stability	
	Ratio	Level (mph)		(ρf)					Min Vol.	1 Wk
	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(%) (10)	(%) (11)
III.19	60/30/10	0.37	-	1.19	0.27	0.32	5	0.39	88	95
III.20	60/30/10	0.34	-	1.07	0.50	0.54	3	0.52	83	94
III.21	60/30/10	0.31	-	1.07	1.65	1.70	1	0.63	83	96
III.22	60/30/10	0.28	-	1.11	1.72	0.80	1	0.67	83	95
*III.23	60/30/10	0.25	-	1.37	1.62	2.22	58	0.86	96	98
III.24	60/30/10	0.25	0.3	1.38	0.30	0.41	5	0.51	85	96
III.25	60/30/10	0.22	0.3	1.41	0.49	0.69	3	0.62	89	95
III.26	60/30/10	0.19	0.3	1.67	0.30	0.50	4	0.61	84	94
*III.27	60/30/10	0.16	0.3	1.96	0.59	1.16	56	0.76	98	98

Notes: *Not an example of this invention

(2) through (11) = the same as in Table II

TABLE IIIb (CONT.)

Test No.	HCFC-142b/EtCl/CO₂ Ratio (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (ρf) (5)	Cell Size (mm) (D) (6)	pf●D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Dimensional Stability Min. Vol. (%) (10)	Dimensional Stability 1 Wk (%) (11)
III.28	60/20/20	0.37	-	1.27	0.27	0.34	8	0.32	93	92
III.29	60/20/20	0.34	-	1.28	0.21	0.27	6	0.28	93	94
III.30	60/20/20	0.31	-	1.40	1.26	1.36	2	0.36	88	94
III.31	60/20/20	0.28	-	1.29	1.32	0.41	5	0.40	85	94
III.32	60/20/20	0.25	-	1.19	1.76	0.90	21	0.77	89	97
III.33	60/20/20	0.25	0.3	1.44	0.25	0.36	6	0.33	89	96
III.34	60/20/20	0.22	0.3	1.55	0.32	0.50	5	0.34	90	97
III.35	60/20/20	0.19	0.3	1.62	0.31	0.50	5	0.52	87	95
III.36	60/20/20	0.16	0.3	1.93	0.41	1.79	7	0.57	91	96

Notes: *Not an example of this invention

(2) through (11) = the same as in Table II

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The data for foams expanded with a wide variety of
blowing agents support the applicability of Equation 1
as a criteria. Note the DCS values for resin I (as
listed in Table Ia) feedstock (Quantum 7300MF) are 1.21
and 1.89 for the F (foamability) values of 1.8 and 2.8,
respectively. Closed cell foams expanded with EtCl or
CFC/EtCl mixtures having more that 70 percent EtCl show
unsatisfactory dimensional stability. Single CFC
blowing agents, CFC-114 and CFC-12, expand dimensionally
stable foams but the cross-sectional sizes of the foams
are small. Although the foam's cross-sectional
capability suffers from the incorporation of CO_2 as a
blowing agent component, the HCFC-142b/EtCl/CO_2 ternary
blowing agent mixture provides substantially closed cell
foams having satisfactory dimensional stability (Table
IIIb).

Optimal foaming temperatures for the CFC-
114/EtCl blowing agent mixture ranged from 125°C to 131°C
for this specific resin. Optimal foaming temperatures
for the CFC-12/EtCl blowing agent mixture ranged from
127°C to 133°C for this specific resin. Optimal foaming
temperatures for the HCFC-142b/MeCl blowing agent
mixture ranged from 130°C to 133°C for this specific
resin. An optimal foaming temperatures for the
CFC-12b/CFC-11 blowing agent mixture is 130°C for this
specific resin. Optimal foaming temperatures for the
HCFC-142b/EtCl/CO_2 blowing agent mixture ranged from
130°C to 133°C for this specific resin.

Example IV

The apparatus used in this example is a one
inch foam extrusion line as employed in Examples I
through III. In the tests of this example, a high melt

strength polypropylene homopolymer (resin F in Table Ia)
was expanded with several new blowing agent systems as
listed in Table IV.

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TABLE IV

Test No.	Blowing Agent Type (1)	Ratio (Mole) (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (pf) (5)	Cell Size (mm) (D) (6)	pf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Dimensional Stability Min Vol. (%) (10)	2 Wk (%) (11)
IV.1	HCFC-142b/n-butane	80/20	0.22	-	1.30	0.60	0.78	11	0.54	100	100
IV.2	HCFC-142b/n-butane	60/40	0.22	-	1.39	0.48	0.67	28	0.46	98	99
IV.3	HCFC-142b/n-butane	20/80	0.22	-	1.44	0.54	0.78	11	0.54	83	96
IV.4	HCFC-142b/n-butane	60/40	0.22	0.02	1.17	0.77	0.90	49	0.91	102	105
IV.5	HCFC-142b/n-butane	60/40	0.22	0.03	1.52	0.60	0.91	42	0.43	40	76
IV.6	isobutane	-	0.22	-	1.22	0.68	0.83	10	0.87	100	103
IV.7	isobutane	60/40	0.22	-	1.21	0.60	0.73	12	0.69	98	103
IV.8	isobutane/HCFC-141b	60/40	0.22	-	1.39	0.44	0.61	10	0.72	99	100
IV.9	isobutane/HCFC-141b	60/40	0.22	-	1.41	0.48	0.68	8	1.01	96	102
IV.10	isobutane/EtCl	60/40	0.22	-	1.31	0.52	0.68	9	0.80	94	102

Notes: *Not an example of this invention

(1) through (10) = the same as in Table II
(11) Volume of 2 week old foam as a percentage of the initial volume measured within about 30 seconds after extrusion

TABLE IV (CONT.)

Test No.	Blowing Agent Type (1)	Ratio (Mole) (2)	Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (ρf) (5)	Cell Size (mm) (D) (6)	ρf•D (pcf mm) (7)	Open Cell (%) (8)	Cross-sectional Area (CM2) (9)	Min Vol. (%) (10)	2 Wk (%) (11)
IV.11	HCFC-124	-	0.22	-	1.47	0.10	0.15	25	0.29	97	98
IV.12	HCFC-124/EtCL	60/40	0.22	-	1.34	0.45	0.60	2	0.57	96	102
IV.13	HCFC-124/isobutane	60/40	0.22	-	1.38	0.37	0.51	11	0.44	96	100
IV.14	HCFC-124/n-butane	60/40	0.22	-	1.41	0.54	0.76	12	0.80	98	101
IV.15	HCFC-124/HCFC-123	60/40	0.22	-	1.25	0.32	0.40	17	0.30	99	100
IV.16	HCFC-123	-	0.22	-	1.32	0.60	0.79	33	0.70	45	81
IV.17	n-butane	-	0.22	-	1.38	0.74	1.02	17	0.70	73	96
*IV.18	HCFC-141b	-	0.22	0.08	1.39	1.35	1.88	86	1.29	99	99
IV.19	FC-134a	-	0.22	GMS 0.5	1.89	0.11	0.21	29	0.24	ND	ND
IV.20	FC-134a	-	0.13	GMS 0.5	2.12	0.11	0.23	23	0.30	99	99
IV.21	FC-134a/n-butane	60/40	0.16	GMS 0.5	2.04	0.12	0.24	30	0.27	98	100
IV.22	FC-134a/EtCl	60/40	0.16	GMS 0.5	1.84	0.28	0.52	7	0.49	100	102
IV.23	FC-134a/HCFC-123	60/40	0.16	GMS 0.5	1.84	0.18	0.33	16	0.25	98	96
IV.24	FC-152a	-	0.16	-	1.66	0.62	0.97	28	0.66	100	100
IV.25	FC-152a/n-butane	60/40	0.16	-	1.36	0.68	0.92	5	0.58	100	101
IV.26	FC-152a/EtCl	60/40	0.16	-	1.42	0.51	0.72	17	0.57	92	101
IV.27	FC-152a/HCFC-123	60/40	0.16	-	1.17	0.62	0.73	13	0.53	95	101

Notes: *Not an example of this invention

(1) through (10) = the same as in Table II
(11) Volume of 2 week old foam as a percentage of the initial volume measured within about 30 seconds after extrusion

NOTES: (CONTINUED)

HCFC-142b = 1-chloro-1,1-difluoroethane

5   HCFC-141b = 1,1-dichloro-1-fluoroethane

HCFC-123 = 1,1-dichloro-2,2,2-trifluoroethane

HCFC-124 = 1-chloro-1,2,2,2-tetrafluoroethane

HFC-134a = 1,1,1,2-tetrafluoroethane

10   HFC-152a = 1,1-difluoroethane

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All blowing agents evaluated in this example produce a foam having a low density, with the greatest density being only 2.12 pcf (33.9 kgh). For the foams made with this resin, having a tan delta value of 1.09,
5   the DCS value of 1.69 is well applicable with the F (foamability) value of 1.8. Except for HCFC-123 (and probably HCFC-141b if a closed cell foam is made with this blowing agent), all the other blowing agents make dimensionally stable foam. The dimensional stability of
10   the polypropylene foams achieved by the hydrocarbon blowing agents and blowing agent mixtures is surprising and remarkable. Typically isobutane requires a permeability modifier to make a dimensionally stable polyethylene foam while n-butane fail to produce
15   dimensionally stable polyethylene foams even when a permeability modifier is incorporated.

       Among single blowing agents, isobutane performs well as indicated by a relatively large cross-sectional
20   size and excellent dimensional stability. HCFC-124 and HCF-134a are marginal as single blowing agents producing foams having very small cell sizes and cross-sections. A small amount of glycerol monostearate (GMS) was added to HFC-134a blowing agent systems in order to enlarge
25   the cell size. The performance of HFC-152a is adequate. Sometimes, as seen in Table IV, mixing in a high solubility secondary blowing agent improves foam performance (cell size, cross-sectional size, open cell
30   content) of these HCFC and HFC blowing agents.

Example V

       In this example, an accumulation extrusion process is beneficially used to produce a large cross-sectional polypropylene foam having a low density and

small cell sizes. In this method an accumulating system is inserted after the extruder and before the die. The accumulating system can be any accumulating vessel, but in this Example it is a pressure chamber of one liter with a regulated nitrogen pressure source. The foamable

5   gell is accumulated in the chamber and pushed out by nitrogen pressure. The extruder is a 1-1/2 inch (3.8 cm) screw type having additional zones for mixing and cooling at the end of the usual sequential zones of

10   feeding, metering and mixing. An opening for the blowing agent is provided on the extruder barrel between the metering and mixing zones. At the end of the cooling zone there is an attached die orifice having an opening of retangular shape. The height, or die gap, of

15   the opening ia adjustable. The width is fixed at 0.25 inch (6.35 mm).

Resin I in Table Ia is fed into the extruder at a uniform rate of 9.3 pounds per hour. The blowing

20   agent mixture injected into the mixing zone is HCFC-142b/EtCl in the percentages and amounts listed in the Table V. The temperature maintained in the extruder zones were 120°C (centigrade) at feeding zone, 190°C at melting zone, 190°C at metering zone and 160°C at the

25   mixing zone. The accumulator and transfer line are maintained at 138°C. The foamable was accumulated and shot out through the die orifice by nitrogen pressure. The results of this Example are in Table V.

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TABLE V

Test No.	Blowing Agent Ratio (Mole) (2)	Blowing Agent Level (mph) (3)	Talc Level (pph) (4)	Foam Density (pcf) (ρf) (5)	Cell Size (mm) (D) (6)	ρf●D (pcf mm) (7)	Open Cell (%) (8)	Dimensional Stability 1 Wk (%) (10)	Dimensional Stability 2 Wk (%) (11)	Dimensional Stability 8 Month (%) (11)
*V.1	60/40	0.28	-	1.04	1.41	1.47	25	ND**	ND	ND
V.2	60/40	0.28	-	1.21	1.13	1.37	4	75	93	98
V.3	60/40	0.28	0.2	1.08	0.77	0.83	2	73	88	96
V.4	72/28	0.28	0.2	1.40	0.54	0.76	4	87	103	103

Notes: * - Not an example of the present invention ** - Not determined

(2) through (8) = the same as in Table II
(10), (11) & (12) Volumes at 1 week, 2 weeks and 8 months as a percentage of the initial volume measured within about 30 seconds after extrusion

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As can be seen in Table V the foams are
substantially closed-cell and within dimensional
stability limits (with the exception of V.1). All four
examples provided good quality foams with large cross-
sections, including some up to 5.9 square inches (37
5   square centimeters). As can also be seen in Table V
dimensional stability is acceptable.

The accumulating extrusion is one of two
preferred processed methods for producing the
10   polypropylene foam products according to this invention.
The other is the coalesced strand foam method. This
high extrusion rate accomplished through the use of an
accumulating extrusion system (AES) permits production
15   of fold-free foam products having large cross-sections.

Example VI

This example illustrates that the technology is
scalable to a large foam extrusion line. The apparatus
20   used in the example is a 3-1/2 inch (8.8 centimeters)
screw type extruder having essentially the same
configuration as the one employed in Example I except
for the die. The apparatus is equipped with a multi-
hole die having 722 holes of 0.041 inch (1.04
25   millimeters) diameter arranged in 38 rows and 19 columns
in a equilateral triangular pattern with 0.125 inch (3.2
millimeters) distance between holes. The operating
procedure of this apparatus is essentially the same as
30   one used in Example I.

In practice, a polypropylene homopolymer (Resin
F in Table Ia) was fed into the extruder at a uniform
rate of approximately 200 lbs/hr (90.8 kg/hr). The
temperatures maintained at the extruded zones were 130°C

at feeding zone, 200°C at melting zone, 230°C at metering
zone and 230°C at mixing zone. An 80/20 by weight
(72/28 by mole) mixture of HCFC-142b/EtCl was prepared
in a weigh tank and injected under pressure into the
mixing zone at a rate of 34 lbs/hr (15.4 kg/hr) which

5   amounted to 17 parts per hundred parts of polymer of
0.188 mph. When the uniform mixture of the
polymer/blowing agent mixture was cooled down to
approximately 155°C, a good foam having fine cell size

10 was achieved. The foam strands coalesced together
leaving little noticeable interstices among them and
resulted in a foam plank of 2.5 inch x 4.5 inch (6.3 cm
x 11.3 cm) cross-section. The foam has 1.27 pcf density
(20.3 kgh), 0.87 mm cell and approximately 13 percent

15 open cells. The foam had a thermal conductivity of 0.28
B.t.u./(hr)(ft^2)(F/in) which compares favorably with
that of an extruded polyethylene foam plank is typically
0.45 B.t.u./(hr)(ft^2)(F/in). With its resiliency and
low thermal conductivity, the polypropylene foam product

20 is not only useful for cushion packaging but also for
insulation applications.

Example VII

25   This example shows that an extruded
polypropylene foam prepared according to the present
invention may be thermoformed to useful articles. The
foaming apparatus used in this example is the same as

30 used Example VII except that a gap-adjustable slit die
of 1.5 inch (38.1 mm) width was installed on the
apparatus instead of a multi-hole die.

        The same polypropylene homopolymer resin as
used in Example VII was fed into the extruder at a
uniform rate of approximately 200 lbs/hr (90.8 kg/hr).

The extruder zones were set at the same temperatures as in Example VII. A 70/30 by weigh (60/40 by mole) mixture of HCFC-142b/EtCl was prepared in a weigh tank and injected under pressure into the mixing zone at a rate of 34 lbs/hr (15.4 kg/hr) which amounted to 17 parts per hundred parts of polymer of 0.20 mph. When the polymer/blowing agent mixture was uniformly cooled down to an optimum foaming temperature of 155°C, the die gap was adjusted to prepare a foam free from prefoaming. At a die gap of 0.049 inch (1.24 mm), a substantially closed cell (open cell content=3.5 percent) polypropylene foam having 1.21 pcf (19.4 kgh) density and 0.55 mm cell size was obtained. The foam had a thickness of 0.79 inch (2.0 cm) and a width of 3.56 inch (8.9 cm) developed some corrugation. Such corrugation is remedied by increasing the extrusion rate.

The demonstration mold for thermoforming purposes was made from a stainless steel beaker having 3-3/8 inch (8.4 cm) diameter. The beaker was perforated on all sides and had a perforated top closure fit inside the beaker. A 3/4 inch (1.9 cm) oak cube is adhered at the bottom center of the beaker. Foam disks of 3-1/4 inch (8.1 cm) diameter were cut out of the foam plank, put in the mold, heated in a convection oven for 3 minutes at 180°C, taken out and pressed by the top closure. Good moldings well reflecting the mold geometry were achieved.

While embodiments of the method and the foam of the present invention have been shown with regard to specific details, it will be appreciated that depending upon the manufacturing process and the manufacturer's desires, the present invention may be modified by various changes while still being fairly within the scope of the novel teachings and principles herein set forth.

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Claims :

1.   An extrusion foaming process for producing
a closed-cell propylene polymer foam comprising the
steps of:

5   (a) feeding a propylene polymer material,
                having a melt flow index less than 10
                decigrams/minute at 230 degrees centigrade into
                a continuous melting device to form a propylene
                polymer melt;

10   (b) feeding a volatile foaming agent into the
                melted propylene polymer;

                (c) uniformly mixing the propylene polymer melt
15   and volatile foaming agent into a mixture;

                (d) cooling the mixture to a forming
                temperature; and

20   (e) discharging the mixture into a zone of
                lower pressure to form the closed-cell
                propylene polymer foam, the process being
                characterized in that the foam is formed under
                process conditions sufficient to provide the
25   closed-cell propylene resin foam with a

foamability characteristic (F) of less than or
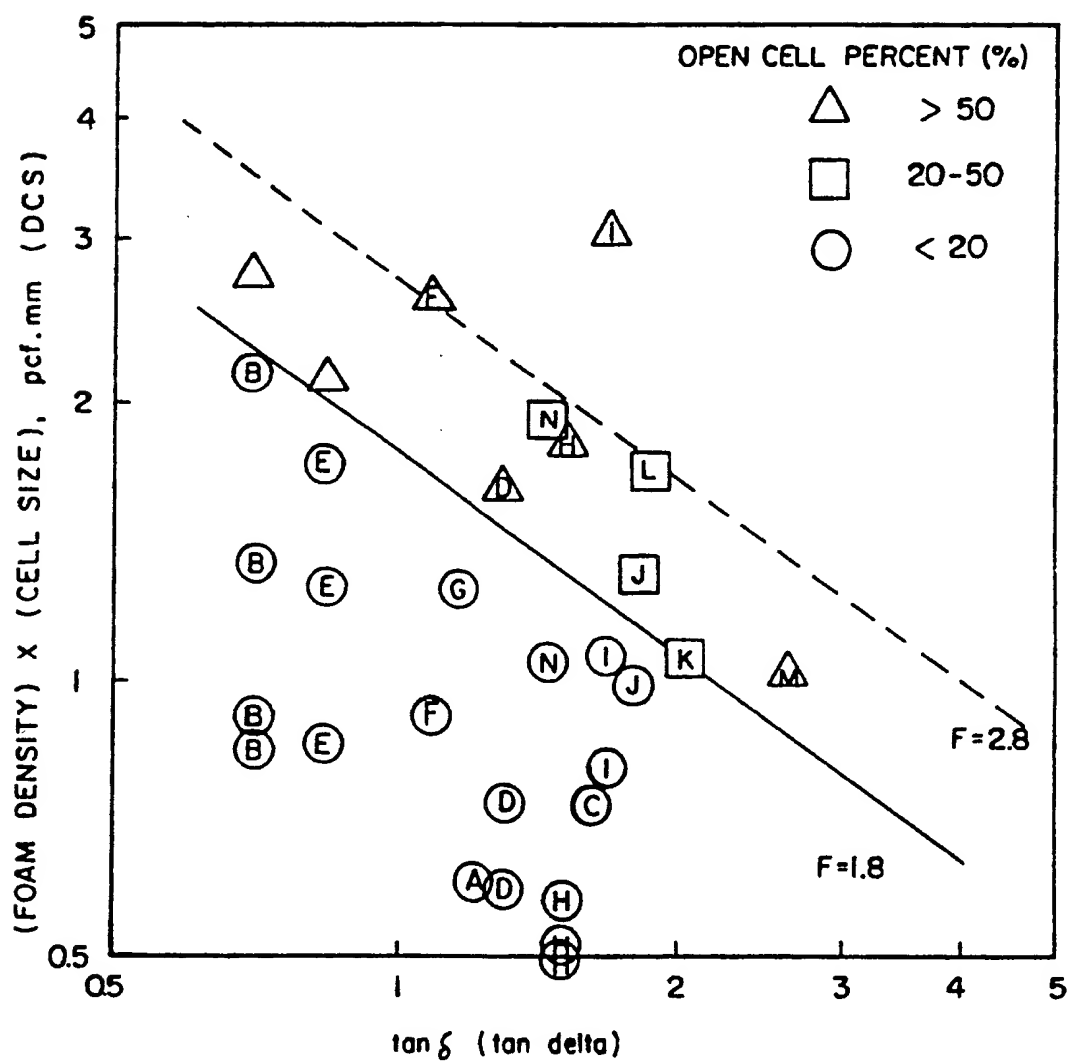equal to 1.8.

2. The process of Claim 1, wherein the
propylene polymer foam has a foamability characteristic
of less than or equal to 1.5.

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3. The process of Claim 1 or 2, wherein the
propylene polymer foam has a minimal cross-sectional
area of at least 5 square inches (32.26 square
centimeters).

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4. The process of Claim 3, wherein the
propylene polymer foam has a minimal thickness dimension
of at least 12.7 millimeters.

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5. The process of Claim 1 or 2, wherein the
propylene polymer foam has greater than eighty percent
(80 percent) closed cells.

6. A closed-cell propylene polymer foam

20 obtainable from the process of Claim 1 or 2.

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FIG. 1

INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :B29C 67/22; C08F 110/06
US CL :264/053; 521/143

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 264/053; 521/143; 264/050, 051; 521/079, 142

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 3,637,458 (PARRISH) 25 January 1972. See column 2, line 48, column 2, lines 71-74, column 3, lines 46-66.	1-6
A	US, A, 3,298,706 (UENO ET AL.) 03 November 1981.	
A	US, A, 4,323,528 (COLLINS) 06 April 1982.	
Y	US, A, 4,424,287 (JOHNSON ET AL.) 03 January 1984. See column 2, lines 48-68, column 4, lines 6-16.	1-6
A	US, A, 4,522,955 (FUKUSHIMA ET AL.) 11 June 1985.	

[X] Further documents are listed in the continuation of Box C.   [ ] See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be part of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
08 MARCH 1993	19 MAR 1993

Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer *M J Miars* ALLAN R. KUHNS
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INTERNATIONAL SEARCH REPORT

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4,714,716 (PARK) 22 December 1987.	
Y	US, A, 4,725,466 (CRASS ET AL.) 16 February 1988. See column 3, lines 4-10.	1-6
A	US, A, 4,824,720 (MALONE) 25 April 1989.	
A	US, A, 3,940,736 (ALTEEPPING ET AL.) 10 July 1990.	
A	US, A, 5,116,881 (PARK ET AL.) 26 May 1992.	
A,E	US, A, 5,180,751 (PARK ET AL.) 19 January 1993.	